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THE EFFECT OF XYLIDINES ON THE STABILITY OF
AN AIRCRAFT-ENGINE LUBRICATING OIL

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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

MEMORANDUM REPORTTHE EFFECT OF XYLIDINES ON THE STABILITY OF
AN AIRCRAFT-ENGINE LUBRICATING OIL

By Walter T. Olson and Emanuel Megrowitz

SUMMARY

Tests were made to determine the effect of xylidines on the stability of an aircraft-engine lubricating oil. The Indiana oxidation test and measurements of the oxygen absorption rate of the oil samples were performed on Navy 1120 oil, Navy 1120 oil plus 0.5 percent by weight of xylidines, and Navy 1120 oil used in piston-ring-sticking tests both without and with xylidines. Laboratory inspections performed on used oils from the beginning and end of several piston-ring-sticking tests without and with xylidines added to the fuel and the oil are compared.

The rate of increase of kinematic viscosity with time in the Indiana oxidation test at 175°C was decreased by the addition of xylidines to new oil. The same is true for used oil with xylidines as compared with used oil without xylidines. The initial rates of oxidation at 175°C , as measured in a closed flask, were less for both new and used oils containing xylidines than for oils containing no xylidines. The addition of 0.5 percent by weight of xylidines to Navy 1120 oil decreased the rate of oxidation of this oil at 175°C as measured in an automatic oil-oxidation apparatus. Comparison of laboratory inspections of oils used without and with xylidines indicated that the xylidines may have had a slight stability-promoting effect. All the oils displayed good stability characteristics in bulk under the conditions of the engine tests. The stability of Navy 1120 oil toward oxidative deterioration, as determined by several laboratory tests, was increased by the addition of small amounts of xylidines.

INTRODUCTION

At the request of the Army Air Forces, an investigation is being conducted to determine the suitability of xylidines as an antiknock component in aviation gasoline. Since it is inevitable that the aircraft-engine lubricant

will become contaminated with fuel, either from blow-by or through oil dilution with fuel during cold-weather operation, tests were performed to determine the effect of xylidines on the stability of engine lubricating oil.

Excessive deterioration of lubricating oil in an engine will not only result in destruction and loss of the lubricant, formation of sludge that may clog oil lines, and formation of corrosive acids, but may also result in deposition of lacquer, ring sticking, and valve sticking. On the other hand, acidic oxidation products resulting from less extensive deterioration may be of real value in improving the load-carrying characteristics of a hydrocarbon oil. Because the principal cause for lubricating-oil deterioration is generally understood to be a process of oxidation, the stability was examined in terms of oxidation tests.

Specifically, the Indiana oxidation test and measurements of the oxygen absorption rate of the oil samples were performed on both new and used oil without and with xylidines. At the beginning and end of the piston-ring-sticking runs, laboratory inspections were made of oil samples removed from the oil system of a single test cylinder of a 12-cylinder liquid-cooled engine.

The tests were performed at the Aircraft Engine Research Laboratory, National Advisory Committee for Aeronautics, Cleveland, Ohio, during June and July 1943.

APPARATUS AND PROCEDURE

Indiana oxidation test. - The Indiana oxidation test apparatus (reference 1) was used to determine the effect of xylidines on lubricating-oil stability. Essentially, the test consisted of blowing air at 10 liters per hour through 300 milliliters of the test oil maintained at 175° C. Samples of approximately 15 milliliters were removed every 24 hours for kinematic-viscosity determination. In addition, Conradson carbon determinations were performed on the original and final samples from the test.

Davis oxidation test. - The initial oxidation rates of the oil samples were determined by the oxygen-absorption method described in reference 2. Briefly, the method consisted of weighing exactly 10 grams of sample into the bottom

of a Sligh oxidation flask, displacing the air in the flask with oxygen, affixing a mercury manometer of about 300-millimeter range to the flask, and immersing the assembly to about the midpoint of the neck of the flask in a constant temperature bath maintained at 175° C. The thermal expansion of the gas caused a pressure increase; the oxidation of the oil caused a subsequent pressure decrease. The pressure was read and recorded at approximately 10- to 15-minute intervals. The drop in pressure from the maximum was recorded as a measure of oxygen consumed at each time interval.

Automatic oil oxidation apparatus. - The oxygen absorption of the samples was also determined in a circulatory system similar to that described by R. W. Dornte (reference 3) but equipped with an automatic recording burette (reference 4). Figure 1 illustrates the apparatus used. Oxygen was circulated through the oil sample by an all-glass pump (reference 5). Absorption tubes containing anhydrous calcium sulfate and sodium hydroxide removed water and carbon dioxide formed during the oxidation reaction.

As oxygen reacted with the oil sample, the pressure in the circulating system decreased. This decrease in pressure caused the control manometer, acting through an electronic relay, to permit the leveling-bottle drive to raise the leveling bottle until the pressure was restored in the system. The position of the leveling bottle was continuously recorded on a drum which rotated at a constant speed. By appropriate calibration and correction for temperature and pressure, the leveling-bottle position was converted to milliliters of dry oxygen absorbed at standard temperature and pressure (0° C, 760 mm Hg). Thus, a record of rate of reaction with oxygen under controlled conditions was obtained.

Test Conditions

| | |
|--|------------|
| Oil sample, grams | 50 |
| Bath temperature, °C | 175 |
| Partial pressure of O ₂ , mm Hg | 720 to 730 |
| Rate of gas circulation, ml/min | 190 |
| Catalyst added | none |

Laboratory inspection. - Used oils from the beginning and end of the piston-ring-sticking test runs were inspected for neutralization number, Conradson carbon, and kinematic viscosity.

Test Specimens

Navy 1120 lubricating oil and Navy 1120 oil containing 0.5 percent commercial mixed xylidines were tested as new oils. The used oil samples were taken from a single cylinder from an engine mounted on a GUE crankcase operated in a program to determine the effect on piston-ring sticking of xylidines added to the fuel and oil.

RESULTS AND DISCUSSION

Indiana oxidation test. - Figure 2 presents the kinematic viscosity of the test samples as a function of the time in the bath. Table I presents the mean values of the Conradson carbon determinations for samples at the beginning and at the end (170 hr) of the Indiana test. The magnitude of the increase in kinematic viscosity and Conradson carbon may be assumed to indicate the approximate extent of oil oxidation.

A comparison of the data for the Navy 1120 oil with the data for the Navy 1120 oil plus 0.5 percent xylidines shows that the addition of the xylidines increased the stability of the Navy 1120 oil under the conditions of the test. A similar comparison between the data for the used Navy 1120 oil and the used Navy 1120 oil with xylidines shows the same effect.

TABLE I

CONRADSON CARBON RESIDUES OF INDIANA TEST SAMPLES

| | Start of test | End of test | Increase |
|---|---------------|-------------|----------|
| Navy 1120 oil | 0.54 | 2.31 | 1.77 |
| Navy 1120 oil plus 5-percent xylidines | .53 | 1.84 | .53 |
| Used Navy 1120 oil (series 11) | .29 | 3.00 | 2.71 |
| Used Navy 1120 oil (series 12) | 1.05 | 3.22 | 2.17 |
| Used Navy 1120 oil with xylidines (series 13) | 1.26 | 2.73 | 1.31 |

Davis oxidation test. - In figure 3 the drop in pressure from the maximum is plotted against time elapsed for the samples. Since the extent of oxidation occurring during the

entire test is small, the curves represent the initial rate of oxidation for the samples of unused oil. Only about 150 milliliters (at 0° C and 760 mm Hg) of oxygen per 100 grams of sample are consumed by the oil during the entire test. Each curve is a mean of at least three determinations. A comparison of the data for the new oils with each other and a comparison of the data for the used oils with each other show that the xylidines inhibited the rate of oxidation of the oils. A comparison of the data for the new oils with the data for the used oils shows that the lubricating oils underwent loss of stability when used in the engine, possibly through beginning oxidation and reception of pre-oxidation catalytic materials from the engine.

Automatic oil-oxidation apparatus. - The effect of 0.5 percent by weight of xylidines on the rate of oxidation of Navy 1120 oil is shown in figure 4. The extent of oxidation for the entire test was about 10 times as great as in the previously described Davis test. Because the used oils were already partly deteriorated, they were not submitted to this more severe test. The oxidation-rate curves (fig. 4) show that the xylidines inhibited the oxidation of the Navy 1120 oil.

Laboratory inspection. - Table II presents average values for laboratory inspection performed on used oils from the beginning and end of several piston-ring-sticking tests. The increases in the values of the various inspection data with engine running time are presented together with the duration of the ring-sticking tests. Mean values and their probable errors were calculated for the tests with no xylidines and for the xylidine tests.

If the neutralization number and the Conradson carbon are taken as criteria, the xylidines appear to have exerted a slight stability-promoting effect, although the observed effect is small and may merely reflect the shorter engine running times. The erratic viscosities probably result from no attempt having been made to remove possible diluents before measuring the viscosity. All the oil samples indicated good stability characteristics in bulk under the conditions of the engine tests.

Aircraft Engine Research Laboratory,
National Advisory Committee for Aeronautics,
Cleveland, Ohio, August 3, 1943.

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TABLE II

LABORATORY INSPECTION OF LUBRICANT SAMPLES FROM PISTON-RING-STICKING TESTS

| Series | Time | | Conradson carbon, percent by weight | | | Neutralization number, mg KOH per gram of oil sample | | | Kinematic viscosity at 210° C (centistokes) | | |
|---|------|-------|--|------|-----------|---|------|-----------|--|-------|-----------|
| | (hr) | (min) | Beginning | End | Increase | Beginning | End | Increase | Beginning | End | Increase |
| No xylidines | | | | | | | | | | | |
| 12 | 31 | 15 | 0.50 | 1.12 | 0.62 | 0.06 | | | 23.85 | | |
| 16 | 26 | 2 | .55 | 0.98 | .43 | .08 | 0.17 | 0.09 | 23.86 | 23.80 | -0.06 |
| 18 | 18 | 23 | .52 | .84 | .32 | .08 | .12 | .04 | 24.07 | 23.76 | -.31 |
| 19 | 15 | | .52 | .90 | .33 | .08 | .09 | .01 | 24.05 | 23.80 | -.25 |
| 25 | 26 | 9 | .59 | .85 | .26 | .10 | .11 | .01 | 22.75 | 23.82 | 1.07 |
| 26 | 25 | 45 | .49 | 1.00 | .51 | .09 | .09 | .00 | 23.85 | 23.99 | .14 |
| Mean | 23 | 56 | | | 0.43±0.12 | | | 0.05±0.03 | | | 0.12±0.35 |
| 3-percent xylidines in fuel, 0.5-percent xylidines in oil | | | | | | | | | | | |
| 13 | 28 | 13 | 0.54 | 1.26 | 0.72 | 0.06 | | | 23.65 | 24.33 | 0.68 |
| 14 | 21 | 24 | .52 | 1.13 | .61 | .06 | 0.12 | 0.06 | 23.83 | 23.65 | -.18 |
| 15 | 19 | 46 | .61 | 0.86 | .25 | .03 | .09 | .01 | 23.50 | 23.48 | -.02 |
| 17 | 24 | 14 | .55 | 1.00 | .45 | .07 | .07 | .00 | 23.62 | 23.98 | .36 |
| 20 | 14 | 57 | .48 | 0.61 | .13 | .07 | .08 | .01 | 23.95 | 24.25 | .30 |
| 21 | 13 | 52 | .59 | .33 | .24 | .09 | .10 | .01 | 23.85 | 23.82 | -.06 |
| Mean | 20 | 24 | | | 0.40±0.14 | | | 0.02±0.01 | | | 0.18±0.07 |

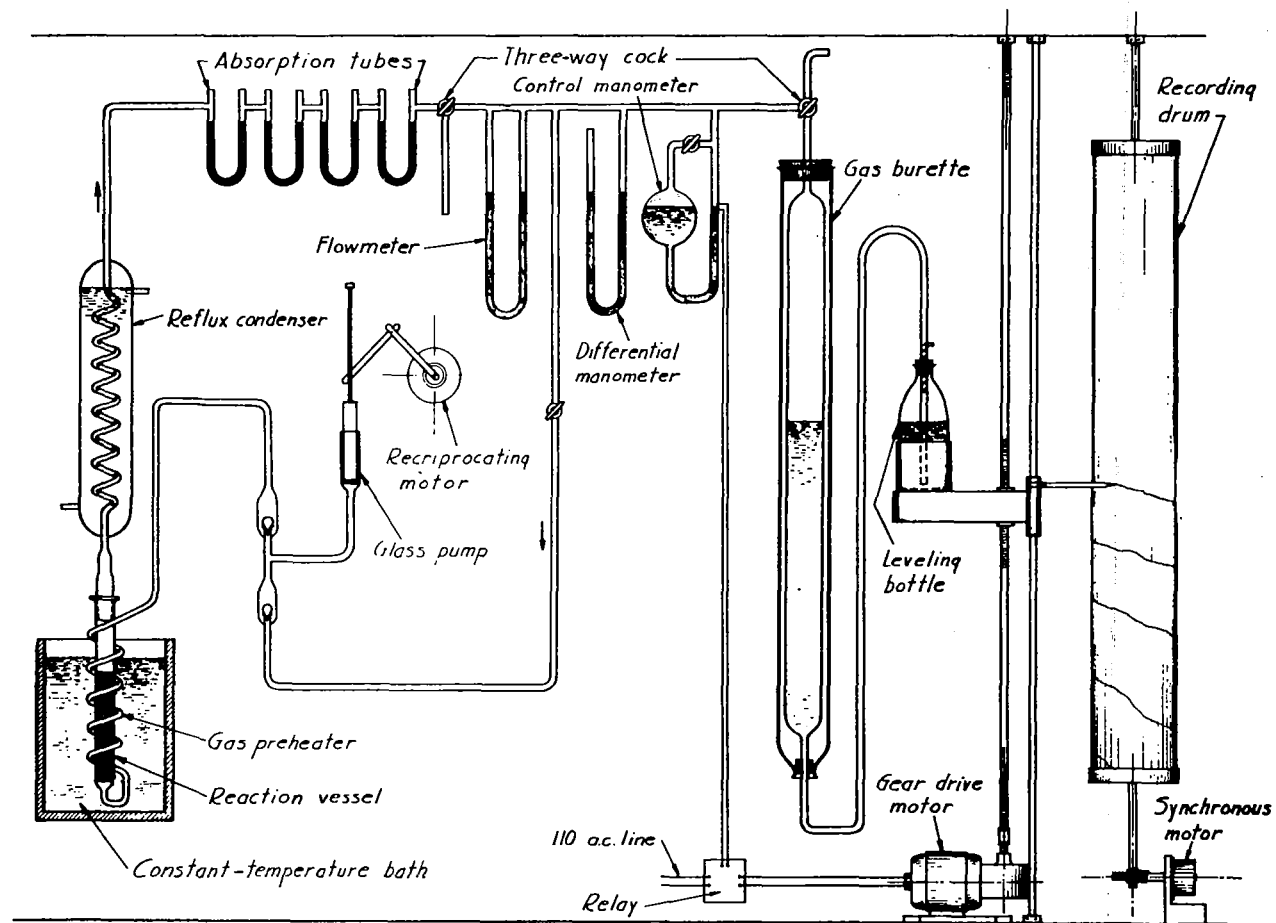


Figure 1.- Automatic oil-oxidation apparatus.

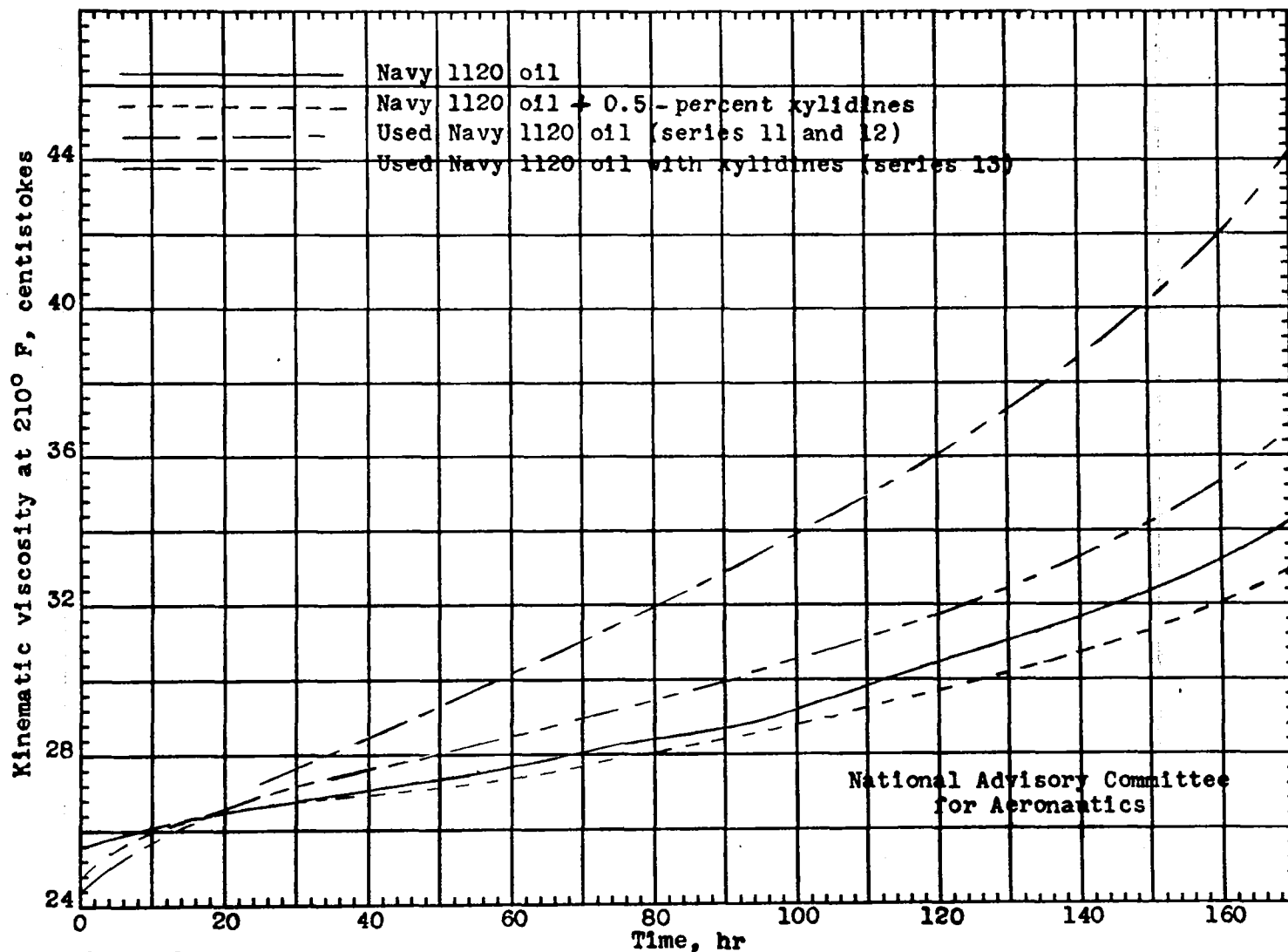


Figure 2. - Kinematic-viscosity increase in Indiana oxidation test. Temperature, 175° C; air flow, 10 liters per hour; 300-milliliter sample; atmospheric pressure.

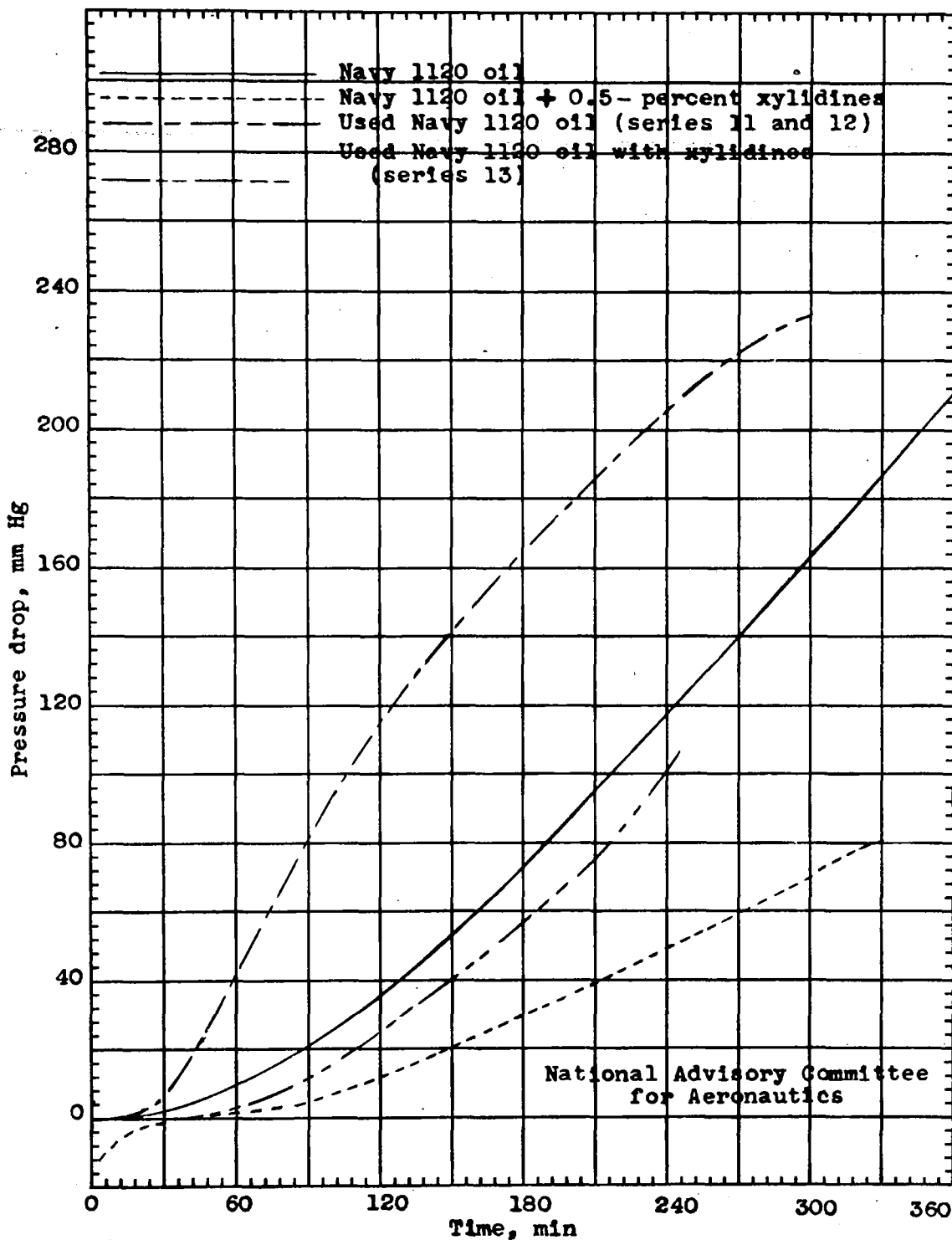


Figure 3. - Effect of xylydines on initial oxidation rates in Davis oxidation test. Temperature, 175° C; 10-gram sample.

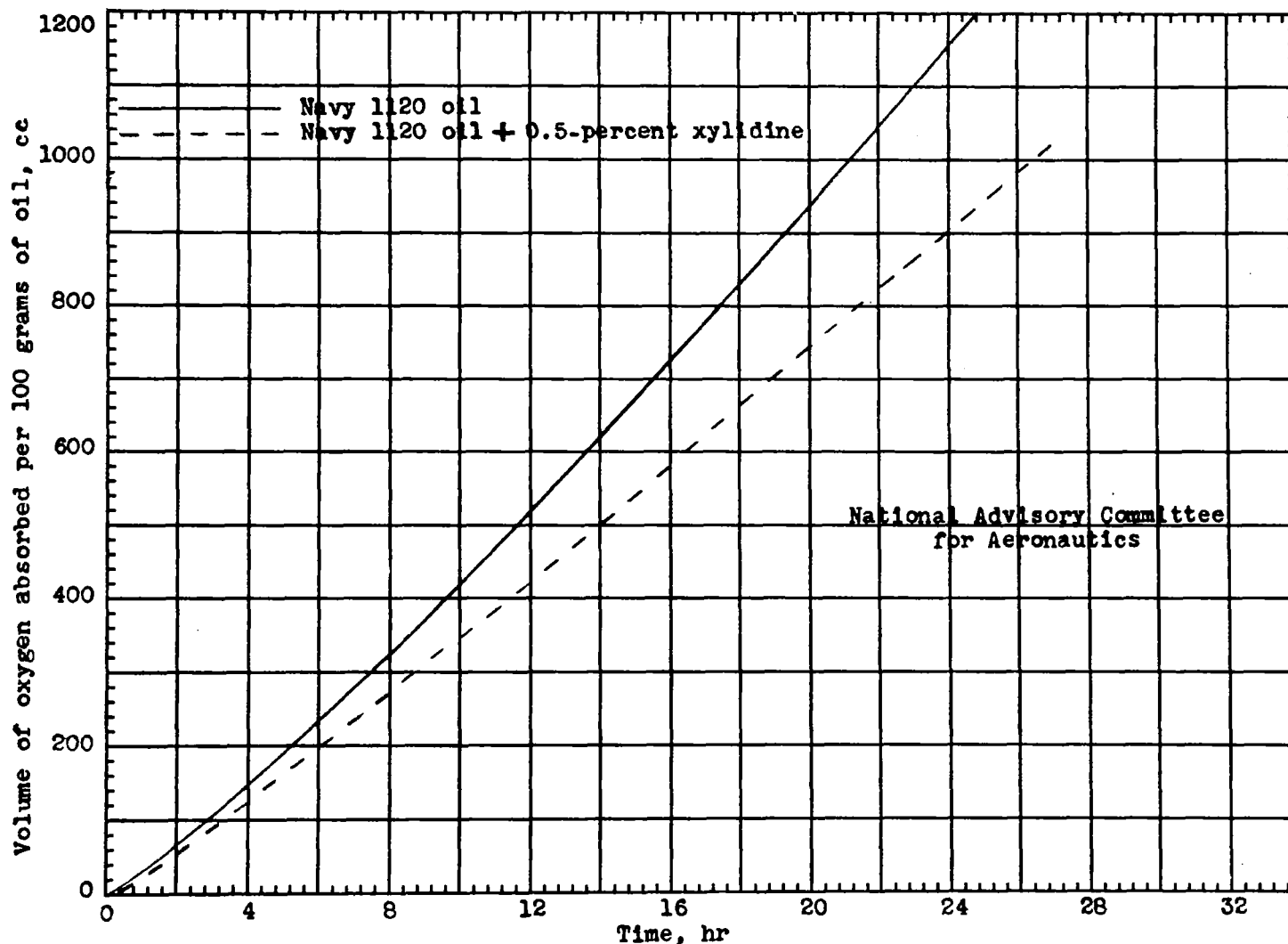


Figure 4. - Effect of xylidines on rate of oxidation of Navy 1120 oil in automatic oil-oxidation test. Temperature, 175° C; circulation of oxygen, 190 milliliters per minute; 50-gram sample; atmospheric pressure. Volume of oxygen absorbed determined for standard temperature and pressure (0° C and 760 mm Hg).

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